Fuel cell electrode has a non-electroplated catalytically active material layer

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Abstract of **DE19757320**

A fuel cell electrode comprises an open porous substrate completely coated with a different catalytically active material. Independent claims are also included for the following: (i) an electrode production process in which a porous substrate is non-electro-coated with catalytically active material; and (ii) a method of producing an electrode/electrolyte unit by depositing amorphous silicon on a solid electrolyte layer, electrochemically etching to create open porosity in the deposited silicon and then immersing in a solution of H2PtCl6 salt in hydrofluoric acid solvent until electroless deposition of platinum on the silicon surface ceases. Preferred Features: The porous substrate consists of silicon and the catalytically active material consists of platinum or platinum-ruthenium.

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The invention concerns an electrode for gas cells as well as a manufacturing process for this electrode. A gas cell exhibits a cathode, electrolytes as well as an anode. An oxidizing agent, e.g. air and the anode are supplied to the cathode a fuel, e.g. hydrogen. Different gas cell types aré well-known, so for example the SOFC gas cell from the block letters DE 44 30 958 c1 as well as the PEM gas cell from the block letters DE 195 31 852 c1. The operating temperature of a PEM gas cell is with approx.. 80 DEG C. At the anode of a PEM gas cell protons form in presence of the fuel with the help of a catalyst. The protons happen to the electrolytes and are connected on the cathode side with the oxygen coming from the oxidizing agent to water. Electrons are set free thereby and electricity is produced. Several gas cells are mechanically interconnected usually for the achievement of large electrical achievements by connecting elements electrically and. An example of such an connecting element represents the bipolar plate well-known from DE 44 10 711 c1. By means of bipolar plates stacked, one above the other electrically gas cells switched into series develop. This arrangement is called gas cell piles. The hydrogen needed for a gas cell can be produced from methanol by an external reformation reaction. With so-called direct methanol gas cell, e.g. methanol admits 0,068,508 B1 electro-chemically directly at the anode in presence of an anode catalyst to protons and CO2 oxidized from EP. The hydrogen-rich gas received by a reformation reaction is among other things contaminated by Carbon monoxide. From the block letters "Journal OF power SOURCES, volume. 29, 1990, side 251" it is well-known that Carbon monoxide the catalyst of the anode poisoned at low temperatures of 80 DEG C sinks accordingly in particular the achievement of the gas cell. The electrically conductive, porous electrode in a gas cell can consist of highly porous carbon, which is mixed with a catalytically active material. As catalytically active material in particular platinmetalle, so e.g. platinum are planned or from platinum ruthenium existing particles. It is from the block letters "M.S. Wilson, J.A. Valerio, S. Gottesfeld; Electrochim. Acta 40 (1995) 355, platinum coal production: K. Kinoshita, P. Stonehart; Preparation and characterization OF highly dispersed electrocatalytic material; Decay Aspects OF Electrochemistry; J.O'M. Bockris, B.E. Conway, eds. Plenum press, NY, volume of 12 (1977) 183" admits to manufacture for PEM gas cells electrodes as follows. Kohlepulver is impregnated with platinum. E.G. for this purpose kohlepulver is in-given to a hexadecimal chlorine platinum acid. Then one reduces. Platinum separates thereby at the kohlepulver. The in such a way impregnated powder is injected together with carbon paper to an electrode.

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This manufacturing process has disadvantageful as a consequence that platinum particles are buried in the coal embedded, in other words. The surface of the electrode is only partly occupied with platinum. Buried platinum does not contribute no more to the electro-chemical reaction in a gas cell. Individual platinum particles at the surface of an electrode are electro-chemically comparatively small active. Generally one tries to maximize the surfaces from catalysts to in order to arrive at good efficiencies. E.g. from the block letters "K. Aika et aluminium, J. Res. Inst. Catalysis Hokkaido Univ., volume. 24, NO. 1, pp. 54-64 (1976)" , therefore the catalytically active material comes out as fine-grained as possible on a substrate is laid on. Particle sizes of 2-5 Nm are usual with electrodes for gas cells. It is also well-known to manufacture a gas cell electrode completely from a platinmetall. So manufactured electrodes are however comparatively expensive, since catalytically active materials are very expensive such as platinum or ruthenium. Task of the invention is the creation of an efficient electrode for a gas cell, with which expensive, catalytically active material is economically used. Furthermore task of the invention is the creation of a manufacturing process for this electrode. The task of the invention is solved by a device with the characteristics of the requirement 1. Furthermore the task of the invention is solved by a procedure with the characteristics of the secondary claim. Unteransprueche represent favourable arrangements. Those would requirement-in accordance with-eat electrode consists of a porous substrate with open porosity. The porous substrate is completely coated with catalytically active material (essentially). By this it is to be understood that also the porenwaende are coated with catalytically active material inside the electrode. The material of the porous substrate differs from the catalytically active material. The material of the porous substrate consists of comparatively inexpensive material such as silicon. In principle all metals are suitable as material for the porous substrate, which are more unedler than platinum and inexpensive. As catalytically active material can be intended platinum or platinum - ruthenium. In the German patent application with the official file reference 197 44 028,2 it is described that the carbon monoxide compatibility of platinum metal particles worsens starting from falling below a diameter Dmin. The diameter correlates with the surface of a particle. It was stated for example that itself the carbon monoxide compatibility of approx.. 10 Nm large platinum particles measurably more badly than of approx.. 15 Nm large platinum particles holds back. A particle size above 15 Nm did not lead no more to (substantial) a change of the carbon monoxide compatibility. This corresponded to the carbon monoxide compatibility of one completely from platinum existing electrode. The carbon monoxide compatibility is therefore a function of the surface of the platinmetalls. A certain minimum

size of the surface is necessary, in order to arrive for a comparatively good carbon monoxide compatibility.

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Dieses Herstellungsverfahren hat nachteilhaft zur Folge, dass Platinpartikel in der Kohle eingebettet, mit anderen Worten vergraben sind. Die Oberfläche der Elektrode ist nur teilweise mit Platin belegt. Vergrabenes Platin trägt nicht mehr zur elektrochemischen

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With the requirement-in accordance with-eaten electrode now a closed large surface of the catalytically active material is present. Thereby the comparatively good carbon monoxide compatibility is guaranteed and a large electro-chemically active surface is made available at the same time. The electrode is particularly efficient therefore. Expensive, catalytically active material is not unused buried at the same time inside the electrode. To the production of the electrode requirement in accordance with a porous substrate is made available, which does not consist of catalytically active material. The substrate is for example cantilever or already on a solid electrolyte layer applied. The catalytically active material is separated afterwards dead at and/or in the porous layer. A dead separation requires favourably no large machine expenditure. Furthermore a dead separation stops automatically, as soon as the porous substrate is covered with catalytically active material. Thus it is on the one hand guaranteed that catalytically active material will not bury no more within the porous body and in such a way to the electro-chemical reaction does not contribute. On the other hand it is reached that the surface of the substrate is coated with catalytically active material completely. For the dead separation of the catalytically active material in particular a redox reaction is planned. For example a porous silicon layer is produced as follows. A so-called wafer is electro-chemically corroded with hydrofluoric acid at the surface. (thin) a porous silicon layer with open porosity, those develops approx. at the surface. 65 volume % pores to exhibit knows. This porous layer is replaced by increase of the current density. The replaced porous silicon layer is cantilever. The catalytically active material and/or a material connection is solved in acid. E.g. it concerns here hydrofluoric acid, in which metal ions are solved. The metal ions come of for example a salt, which was admitted to the hydrofluoric acid. If the porous, into the solution, is immersed cantilever layer then a dead separation takes place. A cause for this is a redox reaction running off. The surface of the substrate is dissolved. The redox reaction stops, as soon as the surface of the porous substrate is covered with the catalytically active material. In a favourable arrangement of the procedure amorphous silicon in or reciprocally, in particular thinly is separated on a solid electrolyte layer of a gas cell. As separation procedure e.g. CVD knows (Chemical vapor deposition) or MBE (Molecular Beam epitaxy) to be planned. Subsequently, by electro-chemical corroding porosity in both amorphous silicon layers is produced. It is to be made certain naturally that the solid electrolyte layer is not soluble in the acid. In particular by-fluoridated connections are soluble not in acid and are suitable as material for the solid electrolyte layer. Nafion TM is a suitable by-fluoridated material. Thin electrode layers are desired, in order to keep for example the materials consumption small. Subsequently, the surface of the porous

silicon layers is dead coated with catalytically active material, until the reactions running off stop. The surface is then covered with the catalytically active material. Cantilever substrates from porous silicon can be manufactured among other things by electro-chemical corroding of silicon under suitable galvanostatischen conditions in simple way. For example in the surface a porous layer can be produced for out single-crystal silicon of existing wafers. Typical current densities for the production of a porous layer lie between 20 and 200mA/cm. The current density, which leads to the separation of the porous layer of the wafer, amounts to approx. 1000mA/cm. The hydrofluoric acid concentration of the etching solution amounts to typically 25 volume %. On such substrates 3n/2 F can be applied + n/4 SI - n/4 SiF6 + ME by dead separation from Metallionen haltiger hydrofluoric acid a catalyst metal, as for example platinum, according to the reaction ME +. For this the substrate from porous silicon is inserted into an aqueous solution, which contains hydrofluoric acid, a wetting agent and a metal salt or a mixture of several metal salts. By dead metal separation the porous substrate (in other words the silicon stand) with a layer of metal becomes encrusted. Thus a porous structure with a metal coat is received. The porous structure with the metal coat can be used as gas diffusion electrode. Remark example A porous substrate of 5 mu m thickness, consisting of silicon, was inserted consisting into a solution of H2PtCl6 and diluted hydrofluoric acid. The dead separation by metallic platinum was to be recognized by a discoloration of the substrate from at first brownish-yellow over grey after black. After the black coloration the substrate in water was washed and presented on a gold substrate. The characterisation of the substrate with separated platinum took place via cyclic Voltametrie in 0,1 M HClO4. The voltage characteristic of the substrate, represented in the figure, showed the typical characteristics of one completely made of platinum existing electrode with large surface. From the hydrogen adsorption charge the specific surface of the catalyst layer can be measured to 100 m /g. The figure shows the cyclic Voltamogramm of the platinierten porous silicon substrate in 0,1 M HClO4. The current density is standardized on the geometrical surface of the diaphragm (sweep guess/advise = potential feed speed, i.e. dU/dt in mV/s). ----------

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Bei der anspruchsgemässen Elektrode liegt nun
eine geschlossene grosse Oberfläche des
katalytisch aktiven Materials vor. Hierdurch
wird die vergleichsweise gute
Kohlenmonoxidverträglichkeit sichergestellt
und zugleich eine grosse elektrochemisch
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1. Electrode for a gas cell, comprehensively a porous substrate with open porosity, whereby the porous substrate is coated with catalytically active material completely and differs the material of the porous substrate from the catalytically active material. 2. Electrode with the characteristics of the preceding requirement, with which the material of the porous substrate consists of silicon. 3. Electrode with the characteristics after one of the preceding requirements, with which as catalytically active material platinum or platinum ruthenium is intended. 4. Electrode with the characteristics after one of the preceding requirements with a thickness of the electrode of less as 50 mu m, in particular less than 5 mu m. 5. Procedure for the production of an electrode, as a porous substrate is dead coated with catalytically active material. 6. Procedure for the production of an electrode electrolyte unit with the following steps: - amorphous silicon is separated on a solid electrolyte layer, - in the separated silicon by electro-chemical corroding an open porosity is produced, - the solid electrolyte layer with the porous silicon layer is inserted into a solution with the salt H2PtCl6 and the solvent hydrofluoric acid, until the dead separation of platinum at the silicon surface, using then, stops. -

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1. Elektrode für eine Brennstoffzelle,
umfassend ein poröses Substrat mit offener
Porosität, wobei das poröse Substrat
vollständig mit katalytisch aktivem Material
beschichtet ist und sich das Material des
porösen Substrates vom katalytisch aktiven
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